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Studies of Organic Pigments by Electron Microscope

AUTHOR(S):

Suito, Eiji; Uyeda, Natsu

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faces on deposition and crystallization. The Sample B was composed of secondary particles of fine elementary particles which were $80\text{ m}\mu$ in diameter. Sample C, Zn, contained very thin hexagonal crystals of 1-2 microns in size among finely dispersed particles. Sample A, Pb, contained thin oblong particles whose mean particle size was $210\text{ m}\mu$ in length and $48\text{ m}\mu$ in width.

We also investigated on the characters of these particles with electron diffraction method. The diffraction patterns obtained by ordinary apparatus gave us n-patterns and p-patterns, showing Debye-Scherrer rings of finely divided particles. Calculation gave us, through the agency of standard diffraction pattern of evaporated gold, the interplanar distances (d) of the fine crystalline metal particles. The analysis of these series of d suggested us that sample A, B and C were PbO , Cu_2O and pure Zn respectively. When Sample B was exposed to the air for about 10 days the diffraction pattern varied from Cu_2O to CuO , and it was inferable that the oxidation had taken place in atmosphere. The symmetry of the n-patterns which were obtained for sample C, showed us that Zn particles had grown up towards the (110) plane which was perpendicular to the c-axis of the crystal structure.

By the above mentioned investigations it became clear that when ultrasonic wave was applied to the system of a solution of electrolyte and reducing metals inserted in it, stripping and dispersing action took place at the reducing metal surfaces, and except for the case that the acidity of the solution was fitted to evolve hydrogen gas from the inserted metal surfaces, oxidation of the dispersed particles will take place by the subsidiary action of the ultrasonic wave, and for some metals this method is useful to prepare microcrystals which have various utilization for study of the "Electron-micro-diffraction method".

11. Studies of Organic Pigments by Electron Microscope

Eiji SUITO and Natsu UYEDA

(Suito Laboratory)

The particle sizes of organic pigments used for the so called "Pigment Resin Printing", whose utility has shown much progress lately, have been attracting much interests of many investigators because of their bright printing effects. Some investigators reported that the particle sizes of organic pigments were of a degree of a few microns, but they measured them rather indirectly. The present investigation was carried out in relation to their true particles sizes directly with electron microscope, and many other interesting results were obtained. The pigments used for the investigation

are as follows: (1) Cyanine Blue Shade (2) Sherdye Blue GFC (3) Aridye Green FJB (4) Aridye Red LDB and (5) Sherdye Orange RC.

They were all preserved in a state of paste, and could be dispersed to colloidal state in such mediums as benzene, toluene, and so on. The specimens for electron microscope were prepared from such colloidal suspensions ultrasonically dispersed in pure benzene. About 50 sheets of electron micrographs were taken in the degree of magnification from 3,000 to 4,500 on the fluorescent screen, and the particle sizes were measured on the enlarged photographs. The size distribution curves were obtained from these data, measured on about 200 particles with respect to each sample. Calculations gave us the mean diameters d_m , the deviations from the mean value, δ , and the standard deviations (S.D.). These results are summarized in Table 1.

Table 1.

No.	Pigments	Dia. ($m\mu$)	d_m ($m\mu$)	$\pm\delta$	S.D.	Shape
1	Cyanine Blue Shade	<i>l</i> : 60-300 <i>w</i> : 16-115	140 52	43 19	29.3 35.8	Diamond like thin
2	Cyanine Blue (elongated)	<i>l</i> : 3000-4000 <i>w</i> : 300	— —	— —	— —	Band like thin
3	Sherdye Blue GFC	<i>l</i> : 30-140 <i>w</i> : 8-35	84 19	20 7	23.3 34.6	Feather like thin
4	Aridye Green FJB	5-200	14	—	—	Ambiguos very thin
5	Aridye Red LDB	<i>l</i> : 35-500 <i>w</i> : 15-100	280 53	92 17	32.3 32.8	Bamboo leaf thin
6	Sherdye Orange RC	3-80	41	10	22.2	Oblong

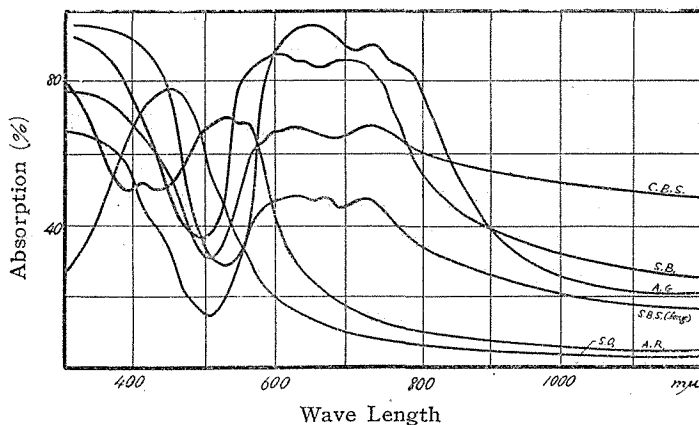
l: length, *w*: width

The results obtained showed us that these particles of organic pigments were all smaller than one micron even in their long diameters, and such a pigments as Aridye Green could be dispersed into a perfect colloidal state whose diameters were within the degree of 50 millimicrons.

The particles of Cyanine Blue shade which are composed of the molecules of copper phthalocyanine (same as Sherdys Blue), grew up in the suspension medium of pure benzene from the small diamond-like particles to oblong band-like particles of 5-10 microns in length. The polymorphologic transition was reported by some investigators, but in these cases the transition took place under the bombardment of electron in the microscope. The transition newly discovered by us differed from the above mentioned, because the transition took place in the suspension at room temperature, and this could be proved with an ordinary optical microscope, because it can resolve the particles of such a degree of dispersion as 5-10 microns. The mechanism of this phenomenon is not clear as yet.

We also investigated on the absorption spectra of the suspension of these organic pigments with Beckmann spectro-photometer, and the results

were summarized in Fig. 1. The four pigments, (1)-(3) and elongated (1),



which are composed of copper phthalocyanine and its derivative showed similar type of absorption spectra, which had a wide band of absorption between 5500 Å and 8000 Å, and minimum at about 5000 Å. The slight difference between the original (1) and the elongated one is considered to be ascribed to the transition of inner structures of the particles.

12. On the Relation between the Color and the Quantity of Iron in Paper Clay

Eiji SUIITO, Kazuyoshi TAKIYAMA and Hiroshi SHIBANUMA

(Suito Laboratory)

Clay highlights some of the many important advances which have resulted from the advent of synthetic rubber and the rapid growth of the machine coating of paper. So it is necessary to study the chemical and physical properties of clay, among which its size as well as its color plays the most important role. Of these two, the latter alone will here be treated. The color is mostly affected by the quantity of iron, especially in its ferric form. To determine the quantity of iron in clay the gravimetric procedure is generally preferable, but the colorimetric method is used to determine it in a simpler way. Trace of iron is usually determined colorimetrically with thiocyanate as reagent. Since many factors affect the color shades or the color intensity of the red compound formed, the proper procedure and due consideration of possible interferences are necessary to obtain satisfactory results.

Standard iron solution: Ferrous ammonium sulfate was dissolved in water and some sulfuric acid was added, then the solution was oxidized by